

GAU 1621

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicants: Buchwald, S.L. et al.

Examiner: Barts, S.

AUG 30 2001

Application Serial No.: 09/239,024

Art Unit: 1621

TECH CENTER 1600/2900
8/30/01

Filed: January 27, 1999

Atty. Docket No.: MTV-009.02

Title: *Arylation and Vinylation of Activated Carbons*Assistant Commissioner for Patents
Washington, DC 20231

Certificate of Mailing

I hereby certify that this "Declaration Under 37 CFR § 1.131" is being deposited with the U. S. Postal Service as First Class Mail with sufficient postage on the date set forth below in an envelope addressed to:

Assistant Commissioner for Patents, Washington, D.C. 20231.

8/24/01

By: *Dana Gordon*

Date of Signature and Mail Deposit

Dana Gordon

Declaration Under 37 CFR § 1.131

Dear Examiner Barts:

As an inventor named on the above-identified application, I hereby declare that the subject matter of rejected claims 1-35 was invented in the United States prior to the effective date, October 16, 1997, of United States Patent 6,057,456.

In support of this Declaration, I attach hereto copies of pages, with their entry dates redacted, from laboratory notebooks maintained by me or one or more of my joint inventors, establishing a reduction to practice of the subject matter of the rejected claims prior to the effective date, October 16, 1997, of United States Patent 6,057,456. Notwithstanding the fact that the entry dates on these pages have been redacted, I hereby declare that these documents establish that a reduction to practice of the subject matter of the rejected claims took place in the United States prior to the effective date, October 16, 1997, of United States Patent 6,057,456.

I hereby declare that all statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Stephen L. Buchwald:

Stephen L. Buchwald

Date: 8/23/01

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09/239,024

264/66 $\text{C}_6\text{H}_5\text{Br} + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° $\xrightarrow{\text{CuI} + \text{SiO}_2}$ ^{OPPPF} Fukuyama

Control 267 $\text{C}_6\text{H}_5\text{Br} + \text{NaO}^+\text{Bz}$ Toluene, 70° \rightarrow No prod

Control 268 $\text{C}_6\text{H}_5\text{Br} + \text{NaO}^+\text{Bz}$ DMF, 70° \rightarrow No prod

269, $\text{C}_6\text{H}_5\text{Br} + \text{Ph}_2\text{P}^{\text{O}}\text{H} + \text{NaO}^+\text{Bz}$ DMF, 70° \rightarrow $\text{C}_6\text{H}_5\text{P}(\text{O})(\text{OEt})_2 + \text{C}_6\text{H}_5\text{Br}$ 34%

270. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{K}_2\text{CO}_3$ Pd(dba)₃, Tl-Bnyp, Toluene, 160° \rightarrow No prod

271. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{K}_2\text{CO}_3$ Pd(dba)₃, TPPF, Toluene, 80° \rightarrow No prod

272. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Pct₃, Toluene, 70° \rightarrow No prod

273. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° \rightarrow No prod

274. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° \rightarrow No prod - but $\text{H}_2\text{C}_6\text{H}_4$ 28

275. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° \rightarrow No prod

276. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° \rightarrow No prod - but $\text{H}_2\text{C}_6\text{H}_4$ 28

277. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, TPPF, Toluene, 80° \rightarrow No prod

278. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Pct₃, Toluene, 70° \rightarrow No prod

279. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° \rightarrow No prod

280. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° \rightarrow No prod - but $\text{H}_2\text{C}_6\text{H}_4$ 28

281. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, TPPF, Toluene, 70° \rightarrow No prod

282. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70° \rightarrow No prod

283. see above (but NaOAc - 3%)

284. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70°, THF \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 59 (6)

285. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70°, THF \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 10% CCR

286. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 70°, THF \rightarrow No prod

287. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 50° \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 28 (6)

288. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 50° \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 28 (6)

289. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 50° \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 28 (6)

290. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, Tl-Bnyp, Toluene, 50° \rightarrow No prod

291. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, TPPF, Toluene, 50° \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 28 (6)

292. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, TPPF, THF, 25° \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 28 (6)

293. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, TPPF, THF, 25° \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 28 (6)

294. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Pd(dba)₃, TPPF, THF, 25° \rightarrow 28 prod

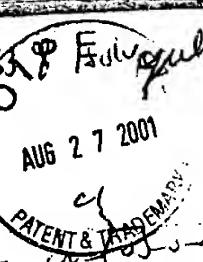
295. $\text{C}_6\text{H}_5\text{Br} + \text{Li}^+ + \text{NaO}^+\text{Bz}$ Tl-Bnyp \rightarrow $\text{H}_2\text{C}_6\text{H}_4$ 65% w/o yield

296. OPPF - no prod

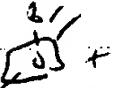
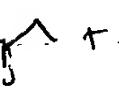
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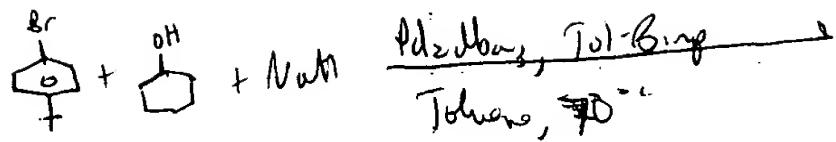
301  +  + NaBH₄, THF, 70° 82% yield

302  +  + NaBH₄, THF, 70°

303  +  + NaBH₄, THF, 70°

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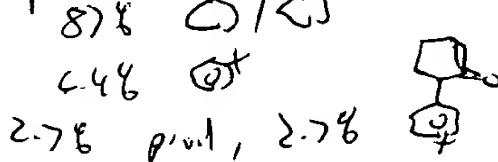
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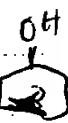
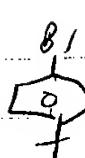
Compound	M.W.	equiv.	mole	amount	volume
t-Buyl Bromohydrin	213.12	1.0	0.5	55 μl	
Cyclohexanol	86.13	5.0	2.5	363 μl	
NaH (60g)	23.94	6.0	3.0	120 mg	
Pd, Phos,	415.7	0.015	0.0075	6.4 mg	
Tolu-Bipy	678.74	0.036	0.0018	12.1 mg	
Toluene				4 ml	

An oven-dried Schlenk flask equipped with a stir bar was charged with NaH, evacuated and back filled with argon. To this was added 3 ml of Toluene and cyclohexanol. The mixture was heated to 70°C for 10 min, at which time t-Buyl Bromohydrin, Pd, Phos, Tolu-Bipy and 1 ml of Toluene were added. The resulting mixture was heated to 70°C while under argon.

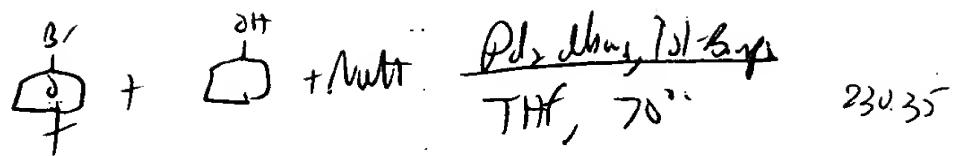
11/26/96 - $\text{off} = 15 \text{ bars, 700 mL aliquot}$
 mp 61-71 - 282-1



MP 61MS-T - 282-1

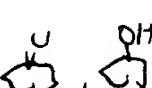
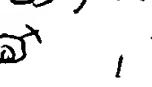


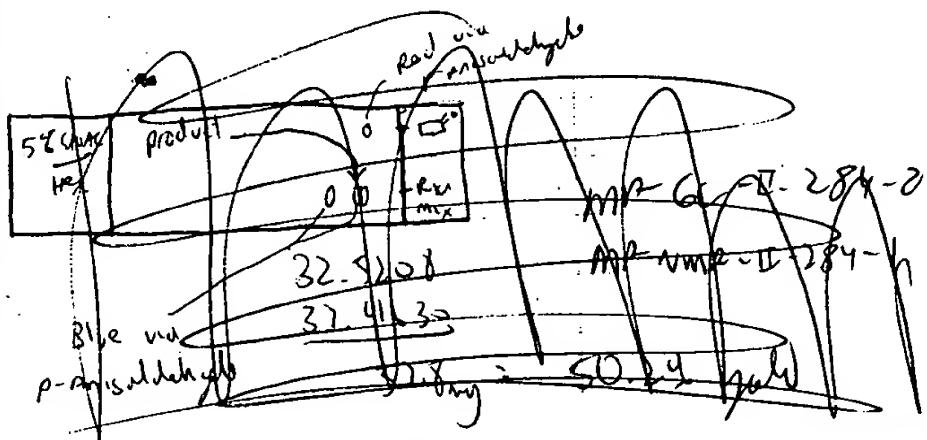
09/239,024



Compound	MW	equiv	mole	equiv
t-Buyl bromoethane	213.12	1.0	0.5	55ml
Cyclohexanol	86.13	1.2	0.6	87ml
NaBH ₄ (6.0g)	23.44	2.0	1.0	40ml
Pd ₂ (Ph) ₄	415.1	0.017	0.0075	6.9mg
Tol-Bnag	678.74	0.031	0.018	12.2mg
Toluene				3ml

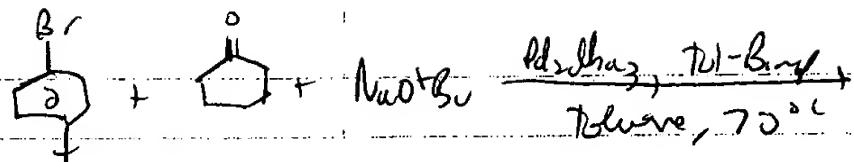
An oven dried Schlenk flask containing a stir bar was charged w/ NaBH₄, evacuated and back filled w/ argon. This was added 2 ml of THF and cyclohexanol. The mixture was heated to 70° for 10 min, at which time t-Buyl bromoethane, Pd₂(Ph)₄, Tol-Bnag and 1 ml of THF were added. The mixture was heated to 70° while under argon.

11/26/91 - after ~20 hr, looks elegant
 : MP CC-II - 284-1 67% 
 : 27%  58% 



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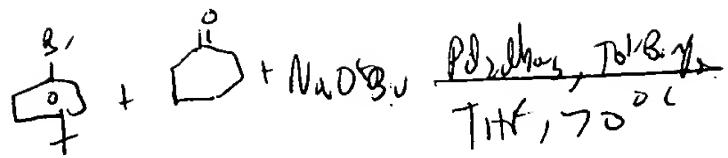
285



Compound	MW	equiv	mole	amount	source
2-Biphenylmethane	213.12	1.0	0.5	55 μl	
Cyclohexane	98.15	1.2	0.6	62 μl	
NaO ₂ Bz	16.7	1.3	0.65	45 mg	
Pd ₂ (Phos)	415.7	0.015	0.0075	6.4 μg	
Tol-2-imp	178.14	0.036	0.008	12.2 μg	
Toluene				3 ml	

An oven dried Schlesch flask containing a stir bar was charged w/ NaOBu (from the bath), Pd-Cl₂, toluene and 2 ml of Toluene (all under Argon). To this was added t-BuOEt/Benzoyl peroxide, cyclohexanone and 1 ml of Toluene. The resulting mixture was heated to 70°C while under argon.

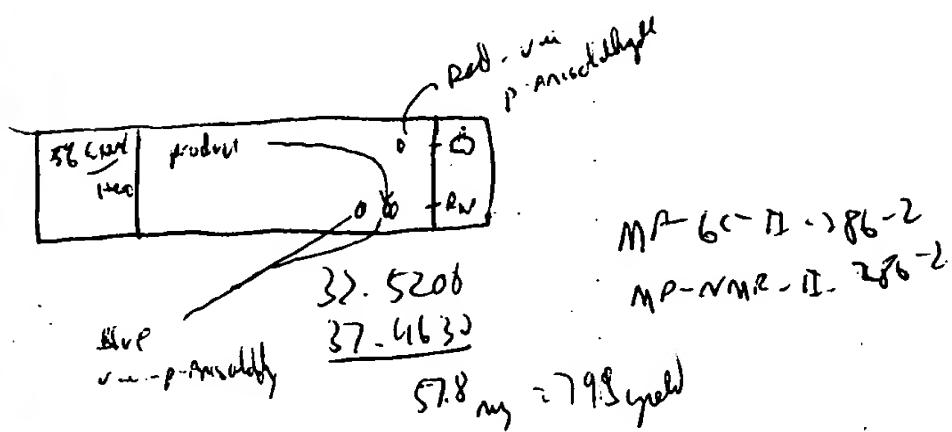
After 6 hours, task elegant MPAC-0-285-1
158  658 P.W. - others

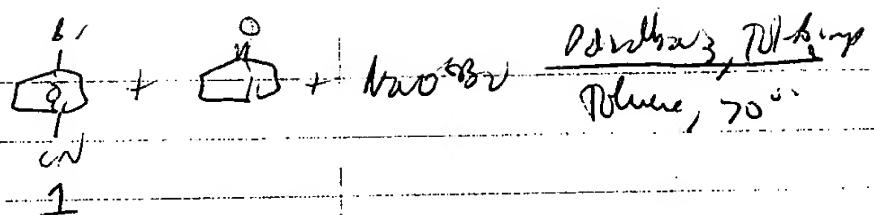


Compound	MW	equiv.	molar (0.31)	amount should be 87 mg	reacs
t-Buyl Bromogene	213.12	1.0	0.31 (0.31)	55 mg	
cyclohexanone	98.15	1.2	0.6	62 mg	
NaO(Bu) ₃	96.1	1.3	0.65	65 mg	
Pd(Phen) ₄	915.1	0.015	0.0071	6.4 mg	
Tol-Bu	678.74	0.031	0.018	1.8 mg	
THF				3 mL	

In oven dried Schlenk flask containing a stir bar was charged of NaO(Bu)(Box), Pd(Phen)₄ and Tol-Bu (under argon). To this was added THF, t-Buyl Bromogene and cyclohexanone. The resulting mixture was heated to 70°C while under argon.

After 6 hr, 70°C eluent - MP 61-71-281-1
238 mg, 73% prod, no sm, no δ



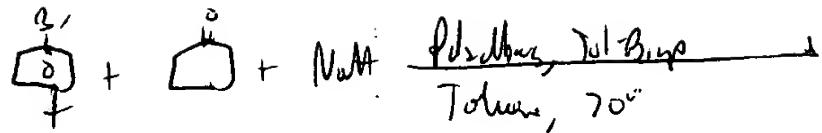


Compound MW equiv molar amount source

4-bromobenzoic acid	182.03	1.0	0.5	91mg	
cyclohexanone	98.15	1.2	0.6	62nd	0.94g
NaO ⁺ Br ⁻ (97%)	96.7	1.3	0.65	65mg	
Tol-Buimp	678.79	0.036	0.018	12.2mg	
Toluene				3nd	
PdCl ₂ Ph ₃	ANAL	0.015	0.0075	6.4mg	

An oven dried Schlenk flask containing a stir bar was charged w/ NaO⁺Br⁻ (8.2g), PdCl₂Ph₃, Tol-Buimp and, I, then 2 ml of Toluene & all under argon. To this was added cyclohexanone and 1ml of Toluene. The mixture was heated to 70° while under argon.

After 4 hours rock absent ANAL GC-II-287-1



Compound	MW	equiv	mole	amount	20ml
t-Buyl bromogro	213.12	1.0	0.1	55 ml	
Cyclohexanone	98.15	1.2	0.6	62 ml	
NH ₃ (60g)	23.99	1.3	0.65	26 ml	
Pd ₂ Na ₂	91.57	0.015	0.0075	6.9 ml	
Tol-Bimp	678.74	0.036	0.018	12.5 ml	
Toluene				3 ml	

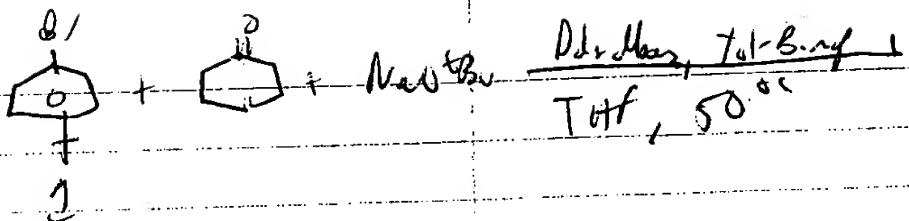
An oven dried Schlenk flask containing a stir bar was charged w/ NaBH₄, evacuated and back filled w/ argon-2. This was added Pd/Catalyst, Tol-Bimp, t-Buyl bromogro, Cyclohexanone and 3 ml Toluene. The mixture was heated to 70° while under argon.

After 6 hours, took aliquot
 MR Col-II-288-1 48% 
 468 sm

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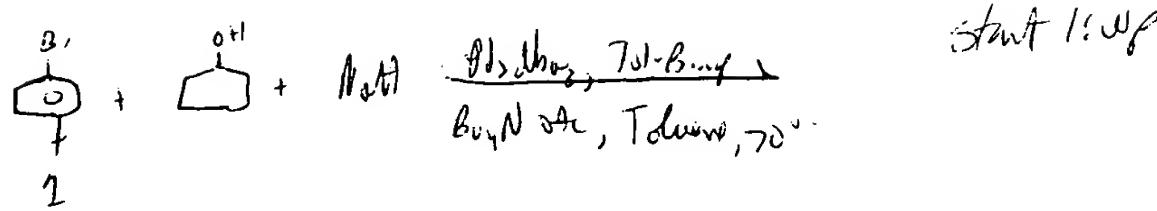
289



Compound	MW	equiv	mole	amount	conc.
1	213.12	1.0	0.5	55 ml	
Cylohexane	98.15	1.2	0.6	62 ml	
NaOBu	96.7	1.3	0.65	65 mg	
Pd/C ₁₀	915.7	0.015	0.015	6.4 mg	
Tol-BuPy	678.74	0.036	0.018	12.2 mg	
THF				3 ml	

An oven dried Schlenk flask containing a stir bar was charged NaOBu, Pd/C₁₀, Tol-BuPy evacuated and back filled w/ argon. To this was added 1, cylohexane and 3 ml of THF. The mixture was heated to 50°C while under argon.

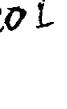
After 3 hours, tank aliquot MP-GC-II-289-1
218 C₂, 8.49 GM, 67.38 prod



Compound	MW	equiv	mol	amount	source
2	213.11	1.0	0.1	55mg	
cyclohexanol	96.13	1.2	0.12	62.4mg	
NaH (60%)	23.94	2.0	1.0	40mg	
Bu ₄ N ⁺ Ac ⁻ (95%)	301.5	1.0	0.5	150.8mg	
D ₂ O, 80%	18.01	0.015	0.0075	6.9mg	
Toluene	678.74	0.030	0.015	12.2mg	
Tetrahydro				3mg	

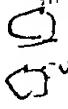
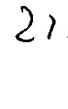
An oven dried Schlenk flask containing a stir bar was charged at NaH, Bu₄N⁺Ac⁻ (in Me₂CO), D₂O, 80%, Toluene and was evacuated and back filled at argon. To this was added 4, cyclohexanol and 3 ml of Toluene. The mixture was heated to 70° while under argon.

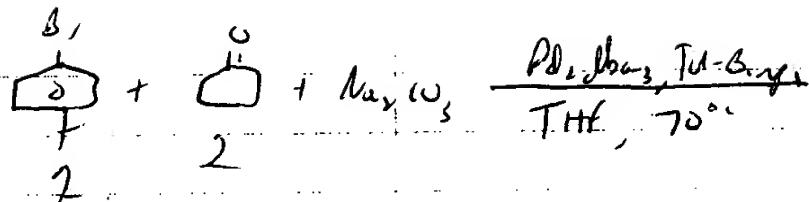
After 3 hours, took aliquot. MP 61-II-240-1

558  201  298 ~~RT = 2.41~~
RT = 2.41

11/30/91

Af 22 h. - Took aliquot MP-61-II-240-1

358  212  378 RT = 2.41



Start 1:00

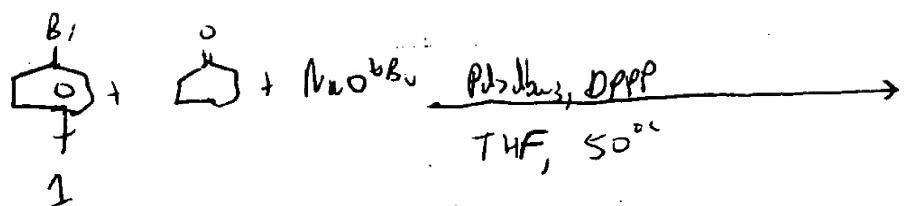
Compound	MW	equiv	mmol	amount
1	213.12	1.0	0.5	55ml
(catalyst) 2	18.15	1.2	0.6	62ml
Na ₂ CO ₃	105.81	1.3	0.65	68.8mL
Pd, Phos	915.7	0.005	0.0025	6.9mL
TD-BuPy	678.71	0.06	0.038	12.2g
THF				3mL

An oven dried Schlenk flask equipped w/ a stir bar was charged w/ Na₂CO₃, Pd, Phos, and TD-BuPy. The Schlenk flask was evacuated and back filled w/ argon. To this was added 1, 2 and 3 mL of THF. The mixture was held at 70° while under argon.

After 3 hours, took aliquot - MP C1-II-291-1

449  488 Sn no prod.

11/20/41 - 8pm = 22 hours, Took aliquot
MP C1-II-291-2 458  488 Sn no prod.



start :wp

Compound	MW	equiv	molar	amount	volume
1 (77)	213.12	1.0	0.5	9.0 ml	
Cyhexaconine	98.15	1.2	0.6	6.2 ml	
NaOEt ₂ (77)	96.7	1.3	0.65	6.5 mg	
Pd ₂ dba ₃	415.7	0.015	0.015	6.4 mg	
OPPP	412.46	0.036	0.018	7.42 mg	
THF				3 ml	

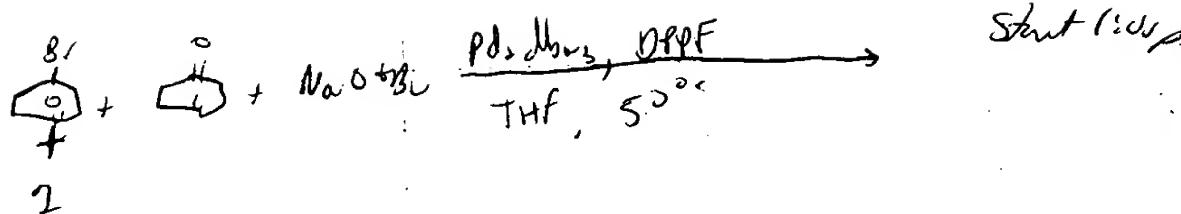
An over dried silicon tube was charged at NaO_2 , $\text{Pd}(\text{Ph}_3)_4$, DPPP, evacuated and back filled at argon. To this was added T, cyclohexane and TAT. The mixture was heated to 50° while under argon.

After 3 hrs, Bkt elegant - MP G II-242-1
♂ 351 ~~55~~, 65 g SM.

12/1/11 - after 24 hr, Test object n/a - II-242-1
158 ♂, 84 ♂, 63 ♀ SM, - 28 y/o.

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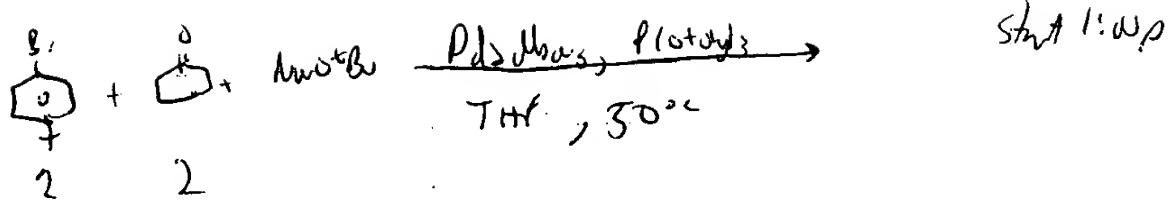


Compound	MW	equiv.	m.mole	amount	volume
I (171)	213.12	1.0	0.5	90 μl	
Cyclohexanone	98.15	1.2	0.6	62 μl	
NaO ⁺ Bu (171)	16.7	1.3	0.65	65 mg	
Pd ₂ Mn ₃	915.7	0.015	0.0075	6.9 mg	
DPPF	554.34	0.03	0.018	10 mg	
THF					3 ml

An oven dried Schlenk tube was charged w/ NaO⁺Bu, Pd₂Mn₃, DPPF, evacuated and back filled w/ argon. Then was added 1, cyclohexanone, THF and the mixture was heated to 50°C tube under argon.

After 3 hours, tube digest mp 61-II-293-1
23% O_2 , 3.5% CO_2 , 46.6% SM, 26% Pd.

12/1/46 - after 24 hours, tube digest mp 61-II-293-2
15.7% O_2 , 19.4% CO_2 , 18% SM, 37.7% Pd.



Compound	M.W.	spur.	amount	amount	source
I (97)	213.12	1.0	0.5	4.0 ml	
Cylohexane	98.15	1.2	0.6	6.2 ml	
NaOBu (97)	96.1	1.3	0.65	6.5 ml	
Pd. dba ₃	415.1	0.015	0.0075	6.9 ml	
P(Ph) ₃	304.37	0.07	0.035	10.7 ml	
THF	50			3 ml	

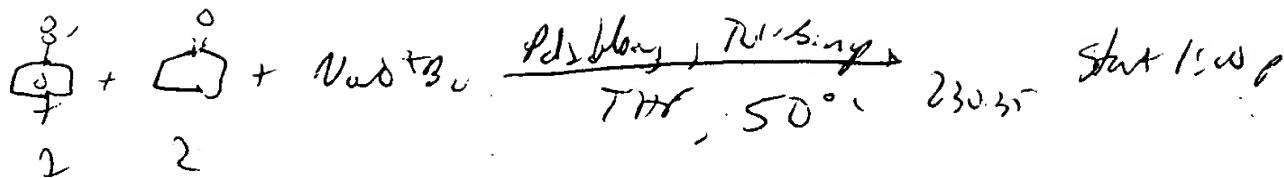
An oven dried Schlenk flask containing a stir bar was charged w/ NaOBu, Pd₂dba₃, P(t-Bu)₃, and was evacuated and back filled w/ argon. 2-Meine was added 2 and 2 w/ THF. The mixture was heated to 30°C while under argon.

After 3 hr, took aliquot - MP 61-II-244-1
6% , 32% , 29% sm, no prod

12/1/96 - after 24 hour, took aliquot - MP 61-II-244-7
5% , 39% , 23% sm, 2% prod.

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295



Compound	MW	equiv.	mole	amount
1 (47%)	213.12	1.0	0.5	10ml
Cyclohexanone	98.11	1.2	0.6	62ml
NaOEt ₃ (47%)	96.1	1.3	0.65	65ml
PdCl ₂ (Ph) ₄	915.1	0.015	0.0075	6.9m
Ti-Bu ₄	678.74	0.25	0.063	12.2mg
THF				5ml

An oven Schlenk flask was charged w/ NaOEt₃, PdCl₂(Ph)₄, Ti-Bu₄ and was evacuated and back filled w/ argon. To this was added 1 and 2, THF and the mixture heated to 50°C while under argon.

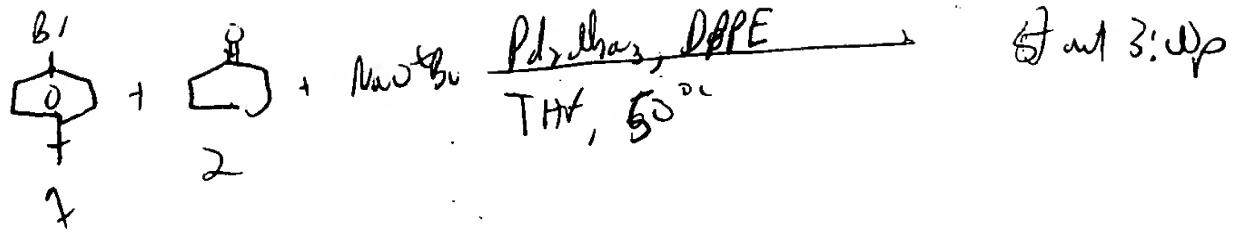
After 3 hrs, took aliquot - mp-66-11-245-1 25° C_2H_5 31% prod.
44% sm no Q/T

12/1/91. 24 hrs, mp-66-11-245-2 7.56 C_2H_5
28 Q/T
86% prod.

46. 4443

46. 3700

$$\frac{46.3700}{74.3m} = 65\% \text{ yield}$$



Compound	MW	equiv	mole	amount	source
1	213.12	1.0	0.1	90nl	
Cyclohexane	98.11	1.2	0.6	62nl	
NaOtBu	96.7	1.3	0.65	63mg	
Pd2BbBr3	915.7	0.015	0.005	6.4mg	
DPPE	551.34 444.34	0.031	0.016	2.1, 2.2 7.2mg	
THF				3nl	

An oven dried Schlenk tube was charged w/ NaOtBu, Pd₂BbBr₃, DPPE and was evacuated and back filled w/ argon. To this was added 1, 2, THF and the resulting mixture heated to 50°C while under argon.

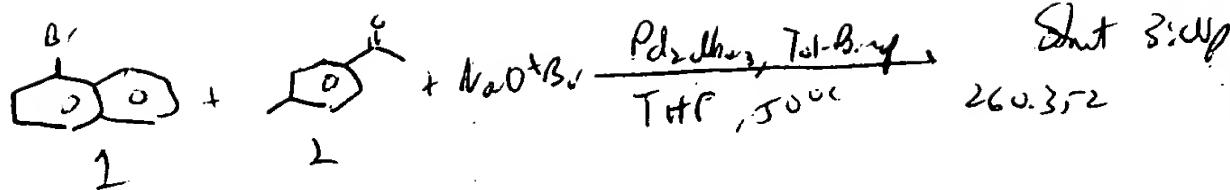
12/2 After 18 hrs - took aliquot MP 61-II-242-1 46 OT
 69g Sm
 No prod.

12/3/92 444.34 - took aliquot MP 61-II-246-2 75 OT
 62g Sm
 No prod.

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301



Compound	MW	equiv	molar amount	source
1	207.08	1.0	0.5	1.0 ml
2	134.08	1.2	0.6	80 ml
NaO ⁺ Br ⁻	96.7	1.2	0.65	65 ml
Pd ₂ dba ₃	415.7	0.015	0.0075	6.4 ml
Tbf-Bn ₃	678.74	0.032	0.018	12.2 ml
THF				3 ml

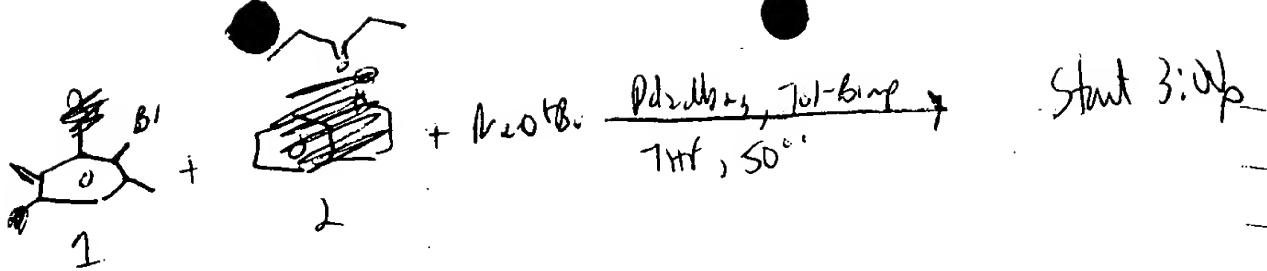
8% naphthalene

12/21/91 - aft = 18 hrs, 70% yield - MP-61-II-301-1 35% SM
44% - RT: 9.603

MP-61-II-301-1 M¹=260

12/31/91 - aft = 24 hrs, 70% yield MP-61-II-2
4% reduced (naphthalene)
29% SM
59% prod RT: 9.642

43.5043
43.3480
106.3 mg 82% yield



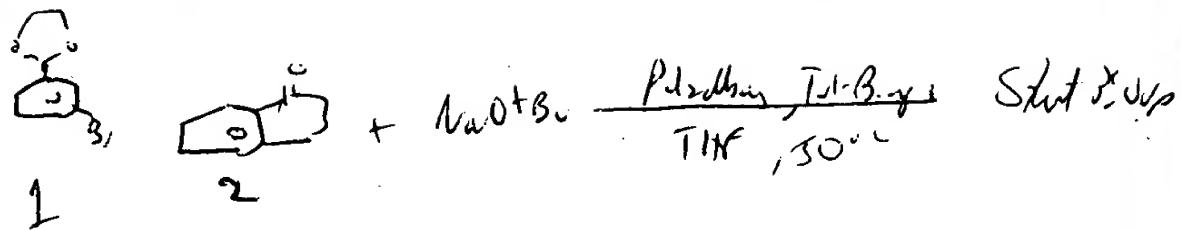
Compound	MW	g/mmol	mmol	amount	
1	185.17	1.0	0.50	67 μl	1.301
2	185.17 86.17	1.2	0.60	64 μl	1.081
NaOtBu	96.7	0.3	0.65	6.5 mg	
Pd2(dba)3	915.7	0.005	0.0075	6.9 μg	
Ts-Bimp	618.79	0.032	0.518	12.2 mg	
THF				3 mL	

¹H NMR (8 hrs - Took aliquot)
 MP-60-II-3021 5% Xylene
 85% SM

¹H NMR After ~44 hours, Took aliquot
 MP-61-5-3022 4.6% Xylene
 82.8% SM

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Compound	MW	mmole	equiv	amount	
1	229.03	1.0	0.5	76 μl	1.51M
2	146.14	1.2	0.6	80 μl	
NaOBu	96.7	1.3	0.65	65 mg	
Pd2Bn	915.7	0.015	0.0075	6.9 mg	
Tl-Bz, imp	678.74	0.032	0.016	12.4 mg	
THF				3 mL	

12/24/96 - after 18h, Took aliquot MP 61-4-303-1
 35° RT = 3.864
 62° RT = 11.26 °

12/31/96 - after 4nh Took aliquot MP 61-4-303-2